CONDUCTIVITY MEASUREMENTS MADE ON LARC-SI AND KAPTON POLYIMIDES FOR SPACE APPLICATIONS

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SUMMARY:

I.   Resistivity measurements performed using the classical conductive electrode method obtained resistivity values of approximately 1E16 ohm-cm. After several months in vacuum, the same measurements were repeated and similar results were obtained.

II.   In the second method, keV electrons in vacuum were applied directly to the exposed insulating surface of the samples. After injecting the electrons, the samples were then monitored for decay of the resulting surface voltage. The RC time constant of the decay indicated that the resistivity was greatly in excess of 6E18 ohm-cm. This method of measurement is more indicative of the charge leakage properties of the polyimides in space.

III.   Two experimental apparatus were developed to generate and monitor surface voltages in the range 10 V to 10 kV. In one apparatus the voltage can be achieved by accumulating electrons that impact the surface with kinetic energies not exceeding 20 eV. The other apparatus causes electrons to impact the surface at greater than 2 keV.

All measurements were performed on samples SI-001 and KA-001 provided by James Bockman and others of NASA Langley.

I. CONDUCTIVE ELECTRODE METHODS

1. Guard Ring Method

Resistivity measurements were made using the copper guard ring deposited onto the sample and applying +/- 64V to the guard ring and measuring the current flowing from the guard ring to the center electrode (copper) thence to ground. Measurements were made at room temperature (25C) and there was no difference in the results when measured with and without room light exposure.
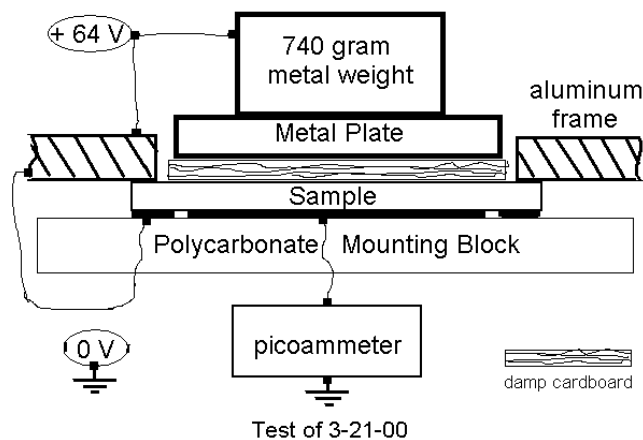

3-20-00 Circuit to measure resistivity from
Guard Ring to Center Electrode
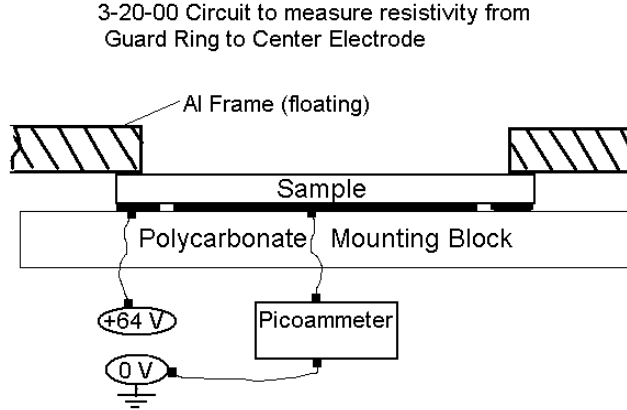
The +64V bias was applied for 10 minutes, and then switched to –64 V. The total change in current was measured to be 0.0012nA for the LaRC-SI sample and 0.0016nA for Kapton. For the moment we will assume that no current flows in the polycarbonate. Using the formula $R = \rho l / A$ one may determine resistivity. The distance from the guard ring to the center electrode is $l = 0.152$ cm. The area through which the current flows in the sample is 15 cm (guard ring circumference) X .0051 cm (sample thickness) = .0765 sq cm. The resistance R = 128V/0.0012nA = 1.07E14 ohms. Similar calculation is performed for the Kapton sample. We find that ρ_{SI}=0.5E+14 ohm-cm for LaRC-SI and ρ_{KA}=0.4E+14 ohm-cm for Kapton.

It is important to note that the current actually does not flow through a constant cross section. One should not expect this method to produce accurate measurements. Some current flows through the polycarbonate. This test simply investigates whether the copper has been sufficiently etched from the surface in the gap region. It has been etched sufficient to our needs so that the current collecting region of the central electrode is well defined.

2. Bulk Method

Resistivity measurements made through the bulk used a damp cardboard contact on the open face of the sample in order to make contact to much of the surface area of polyimide.. A piece of cardboard, dampened with de-ionized water, was held in place opposite the center electrode with a heavy brass weight. The mass of the electrode was briefly varied from 200 g to 2000 g with no effect noted. The cardboard undoubtedly

added ions to the water. Ohm meter measurements found that the resistance of the damp cardboard was always less than one megohm and is thus negligible. +/-64V was applied to the brass weight and the changing currents were measured from the center electrode to ground.

Test of 3-21-00

In a preliminary test the circuit of 3-21-00 was used to measure "total resistivity" within the gap and between the front surface of the sample and the center electrode. The currents in this preliminary test flow both from the guard ring (as on 3-20-00) and through the bulk of the sample from the front surface to the rear surface. In order to estimate the current flowing through the bulk only, one may subtract the guard ring measurements of gap current on 3-20-00 from the measurements of 3-21-00. With 64 volt bias the results of 3-21-00 are: sample SI carried 0.010 nA after 10 minutes settling time, and sample KA001 carried 0.06 nA after 10 minutes settling time. Subtracting the currents of 3-20-00 (typically 0.0006 nA) indicates that most current flows through the bulk, and little flows in the gap.

The measurement is made with the following procedure. Positive 64V bias is applied for ten minutes, at the end of which the current is recorded. The bias polarity is then reversed and after ten minutes the current is recorded. The resistance is defined as the change in voltage (128 volts) divided by the change in current. Based on this ten-minute settle time data we find that (3-21-00):
ρ_{SI}=2.1E+16 ohm-cm for LaRC-SI and,
ρ_{KA}=0.37E+16 ohm-cm for Kapton.
Maintaining the same bias for two hours longer found that the current dropped nearly in half. The current continues to drop for days, but only slightly. Thus, this resistivity value is not applicable for very short or very long exposure to steady bias. Perhaps the drift is due to slow polarization of molecules, rotation of polar molecules, or charge injection at the contacts.

On 3-23-00 the circuit of 3-21-00 was used to evaluate the effectiveness of damp cardboard as well as the change in conductivity after long exposure to bias and to damp cardboard. Tripling the pressure made no change in the current, thus indicating that the wet cardboard was making good "contact" to the front of the samples. Changing the

wetness also made no change. Overnight the current decayed approximately in half for sample KA001. No overnight measurement was performed on sample SI001.

Resistivity measurements on 3-23-00 and 3-24-00 found the following results:
Ten-minute settle time: ρ_{SI} = 1.4E16 ohm cm,
                               ρ_{KA} = 0.34E16 ohm cm.
Eighteen hour settle time: ρ_{KA} = 0.68E16 ohm cm.

The differences between 3-21-00 and 3-23-00 are clearly larger than experimental error and may relate to treatment history of the samples. This is not at all unusual in measurements of insulator resistivity. However, the attachment to the guard ring is improper for good measurement technique and is corrected below. Perhaps some of the data-drift is due to surface currents from the guard ring. These are the measurements made prior to extended evacuation.

3. **Bulk Method After Three Months in Vacuum.**

On 3-24-00 both samples were placed in vacuum at roughly 100 milli-torr where they remained until 6-28-00. The vacuum was maintained at roughly this level by pumping briefly on the chamber about twice per week. The open polyimide surface was directly exposed to vacuum. The samples were then measured again on 6-28-00 to see if vacuum affected their conductivity.

The circuit of 6-28-00 is shown and is the preferred circuit to directly measure bulk material resistivity. It does not have the guard ring current flows that, although small, were present in the data of 3-21-00. The drawing might be misleading because the thickness of the polyimide is greatly enlarged. In reality, the diameter of the damp cardboard sufficiently exceeds that of the copper electrode so that the fringing effects are borne mostly by the guard ring.

The measured currents and resistivities are in the table below. It is interesting that after being evacuated for three months the currents are a little larger than prior to evacuation. We will not speculate about the reasons for this effect. But it must be remembered that the method involves contacts on the samples with the possibility of injection of ions from the contacts, and therefore memory of prior treatments.
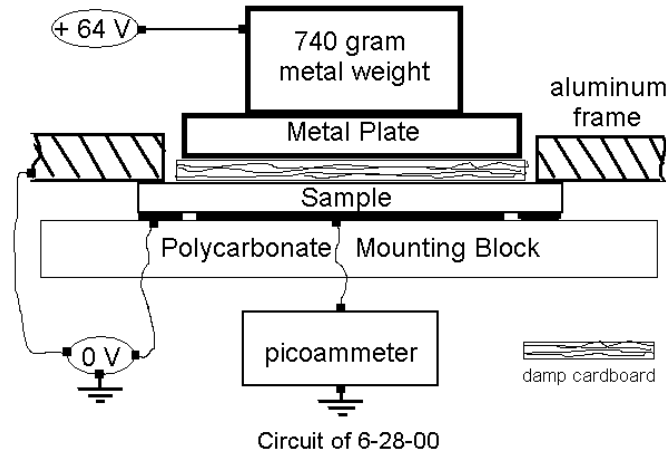
Circuit of 6-28-00

Table of results of 6-28-00.

TIME AT BIAS	SAMPLE SI001	SAMPLE KA001
2 minutes	0.060 nA	0.14 nA
6 minutes	0.030 nA	0.11 nA
10 minutes	0.023 nA, $\rho = 0.88E16$ ohm-cm	0.091 nA, $\rho = 0.23E16$ ohm-cm
4 hours	0.010 nA, $\rho = 2.0E16$ ohm-cm	
7 hours	0.011 nA	
43 hours		0.057 nA, $\rho = 0.37E16$ ohm-cm
123 hours	0.0076 nA, $\rho = 2.7E16$ ohm-cm	

II. KeV ELECTRONS IN VACUUM METHOD.

In order to measure resistivity under spacecraft conditions where non-metalized surfaces of polyimide are exposed to vacuum one uses an electron beam to charge the sample. It is also high-energy electrons in space that charge the sample. Once charged, the surface of the sample can discharge to ground through the bulk-conductivity of the polyimide and onto the grounded copper electrode at the other surface of the sample.

The test method is simple. First one charges the surface. Then the surface voltage is monitored using an electrostatic field probe. The decay of surface voltage is related to the conductivity of the polyimide. It is assumed that the decay is a simple exponential decay where τ is the 1/e time decay constant. The decay of surface voltage is related to the resistivity by the equation $\tau = \rho\varepsilon = RC$. The permittivity of the dielectric is ε.

The prior measurements of LaRC SI indicate that its resistivity is of order 1.4E16 ohm cm. Its permittivity is 2.76E-13 Farads/cm (relative dielectric constant is 3.12). Thus, based on the wet cardboard measurements, one expects that $\tau = \rho\varepsilon = 3.86E3$ seconds = 64 minutes. Therefore the experiment is arranged to measure decay constants of order 64 minutes efficiently. One must be certain that surface voltage does not decay by currents passing through the vacuum and, therefore, the measurements are performed in the dark, and without vacuum gages being near the sample.

Second Crossover Point

One must charge the samples with electrons and not overcharge to the point of breakdown. Since the samples are two mils thick, one must not exceed 1-kV on the surface for long periods (500 V/mil). Additionally, one should be concerned to avoid high field conductivity effects and stay well below the level of 1-kV. The wet cardboard method applied 64 volts through the sample, roughly 1/10 of the voltage where high-field conductivity effects begin to be important (500V/mil). High-field conductivity greatly exceeds regular conductivity. Therefore the electron beam tests should stay well below 1-kV on the surface.

A beam of 1-kV electrons is not capable of charging a polymer surface because for every electron incident more than one secondary electron is emitted. In order to charge the surface one must raise the energy above the energy of the so-called second crossover point, above which secondary electron emission is less that the incident particle flux.

The second crossover point was measured in both samples by exposing the samples to electrons (0 - 5,300 keV) and measuring the voltage on the back copper electrode. Back electrode voltage vs. beam energy was plotted to locate the second crossover point. The crossover point was determined to be 1,800eV; above this energy the electrons entering the film are greater than the number being knocked off the surface, and negative charge begins to accumulate.

The second crossover point is measured with the apparatus diagrammed below.
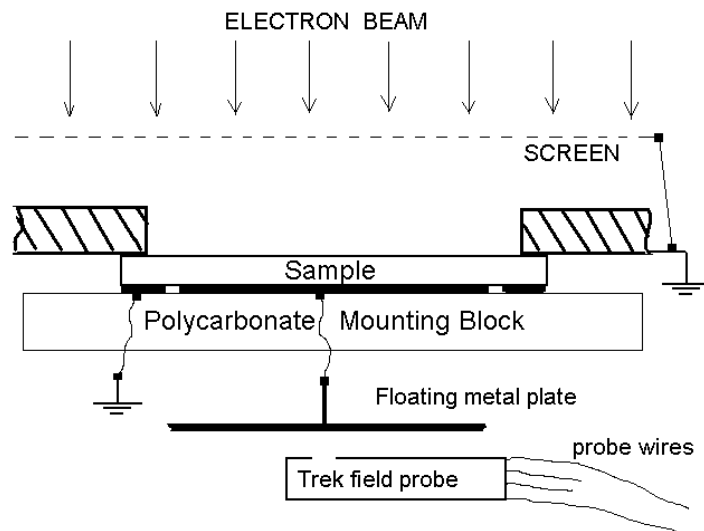
The grounded screen decreases the deflection of the electron beam by the charged sample. This holds the incident electron trajectories so that they are incident upon the sample close to normal (perpendicular to the surface). The field probe is fixed in position near the floating metal plate that is attached to the back copper electrode. The field probe is calibrated by impressing a known voltage on the floating metal plate. For these tests,

the field probe was spaced so that its reading indicated 0.75 Vplate. Thus a 400-volt plate indicated as 300 V on the field probe.

Second Crossover Data
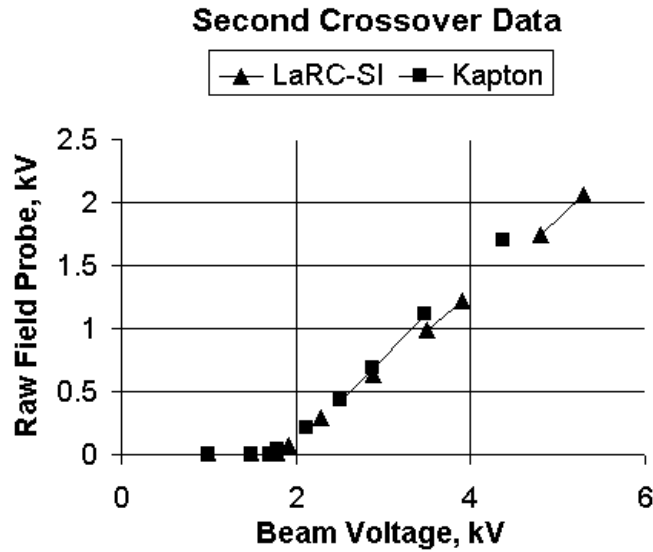
From the second crossover data one sees immediately that the second crossover point occurs at 1.8 kV for both samples. The two slopes are slightly different because the placement of the probe near the Kapton sample was not identical to that of the LaRC-SI sample creating a slightly different calibration factor. The slopes are not equal to 0.75 because the grid did not eliminate the effect of beam deflection by the charged surface.

For this kind of test there is one further calibration factor that needs to be determined. Capacitive voltage division causes the front surface voltage to be different from the back electrode voltage.

Capacitance Model

The following capacitance model represents the electric field probe experimental setup. The field probe monitors the voltage, V_{BE}, on the rear electrode. The field probe voltage was calibrated by applying a known voltage to V_{BE} and reading the field probe. The field probe reading was 75% of the applied V_{BE} in our setup. But when the voltage is caused by a charge, Q, on the surface of the sample, there is a voltage drop in the sample causing a smaller voltage to appear on the back electrode. We need to determine a correction for this effect. The following figure illustrates the effect.

Equivalent Circuit

C_{FS} C_S Q V_S

Q   Sample C_S C_{FS} V_{BE} C_{RS}

V_{BE} plate for probe C_{RS}
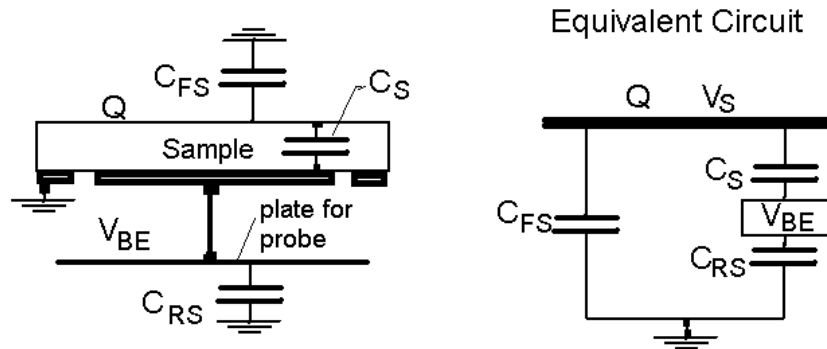
C_{FS} - Front surface capacitance (sample surface opposite the center electrode to mounting hardware and vacuum chamber walls).
C_S - Sample capacitance (front of sample to copper electrode at rear of sample).
C_{RS} - Rear probe plate capacitance (connecting wire and plate to grounded objects including the mounting hardware, guard ring, etc.).
Q   - Charge injected into sample (electrons from electron beam are stopped within the first micron which is essentially on the surface).
V_S - Front surface voltage on sample.

$C_{RS} = 10.5$pF
$C_{FS} = \sim1$-2pF (estimate)
$C_S = \varepsilon A/d = (3.12)(8.85\text{E-}14 \text{ F/cm}) (16.6\text{cm}^2) / (.0051\text{cm}) = 899$ pF
Dielectric constant of LaRC-SI = 3.12
Dielectric constant of Kapton = 3.4

The solution to this system is

$$\frac{V_{BE}}{V_S} = \frac{C_S}{C_S + C_{RS}} = 0.99$$

Thus the capacitance division requires one to make a small correction, only 1%.

Experimental measurement of surface voltage decay.

To measure the time constant for surface voltage decay the sample was exposed to an electron beam at 2.8 kV allowing electrons to enter the sample surface. At 2.8 kV the beam was not capable of charging the surface beyond 1-kV. Current from the rear electrode to ground was monitored during sample charging and measurements. After a charge had built up the beam was turned off and the field probe was moved into position to monitor the sample surface. The probe can be moved from one sample to the other.

Results of Test of Kapton Sample:
The probe had not been calibrated for the spacing from the Kapton sample. But the spacing was similar so that the calibration factor must have been in the range 0.65 to 0.85. At 7/14/00/08:44 (at 25 C) the probe read –247 volts on Kapton and at

7/14/00/11:33 (at 44 C) the probe read –247 volts. Thus the Kapton surface voltage did not measurably decay over a 3-hour period.

Results of test of LaRC-SI sample:
(note: probe zero is offset by -56 volts at the probe)

Date/time (hours)	Probe voltage	actual sample volts
25 degrees centigrade		
7/13/00/17:53	beam off	
7/13/00/17:54	-303	-329
7/13/00/17:56	-303	-329
7/13/00/17:57	-302	-328
7/13/00/17:58	-302	-328
7/13/00/17:59	-301	-326
7/13/00/17:53	-301	-326
7/13/00/18:33	-301	-326
overnight		
7/14/00/08:30	-302	-328
7/14/00/10:30	-303	-329
7/14/00/10:46 begin heating sample		
7/14/00/11:50 sample temp 59 C.		
7/14/00/11:50	-305	-332
7/14/00/12:30 sample temp 81 C		
7/14/00/12:30	-308	-336

Is voltage rise due to probe heating or sample expansion or both??
7/14/00/12:35 begin cooling down from max 83 C.
7/14/00/17:35 25 degrees Centigrade again

7/14/00/17:35	-306	-333

end of test.

A 40-volt calibration test was performed several times during the test by ungrounding the back electrode and applying the 40-V to the back electrode. The measured probe voltage changed by 30 volts, as expected. The probe voltage resolution is +/- 1-volt. Thus the 40-V calibration is good to 3%. As a worst case, assume that the probe drifted by 5% during the 24-hour test. If so, then one may assume that the sample discharged by up to 5% while the probe indicated no discharge. With this assumption we can estimate a lower bound on the time constant and therefore on the resistivity.

Assuming a charge decay of 5% and using the equation: $e^{-t/\alpha} = .95$, when t=24 hours and $\alpha = \rho\varepsilon$,

$-t/\alpha = \ln(.95)$, t = 86,400 seconds, $\ln(.95) = -0.051$
$\alpha = -t/\ln(.95) = \alpha = 1.7E6$ seconds = 19 days.

A charge decay time constant of 19 days is the lower bound on our measurements. The measurements do not exclude an infinite time constant! This time constant implies a sample resistivity, ρ, as follows:

$\rho\varepsilon = (\rho)(8.85E\text{-}14F/cm)(3.12) = 1.7E6$ sec
$\rho = 0.062E20 = 6.2E18$ ohm-cm, or larger.

Such a resistivity is very much larger than the resistivity measured earlier by the classic applied bias method.

III. TECHNIQUE TO CHARGE INSULATORS TO HIGH VOLTAGE BY 10 EV ELECTRON IMPACT.

In August an apparatus was constructed to both control temperature (-80 to +100 Centigrade) and develop HV surface charging by electrons that impact the surface with only ten eV kinetic energy. The first test easily charged a small kapton sample to –300 V. This apparatus may be of interest in the event that one is concerned with possible damage (or with injection of excess mobile electron-hole pairs) introduced by keV electron impact. The method is simple and is analogous to the corona charging method exploited and published by B. Gross and coworkers. The method is now available in our laboratory.

CONCLUSIONS

When a material is used in space applications the material properties appropriate for that environment must be taken into account. Contrary to this advice, resistivity measurements for insulators are usually performed in the earth atmosphere, 1 atm, 20C, with some humidity present and with electrodes attached to the sample. We have repeated such atmospheric tests to find resistivity in polyimide of order 1.4E16 ohm-cm. Such a resistivity predicts a charge decay time constant of $\rho\varepsilon = (1.4E16$ ohm-cm$)(8.85E\text{-}14F/cm)(3.12) = 64$ minutes. Thus, charging by space radiation may be incorrectly assumed to quickly dissipate.

Values of various magnitudes are commonly assumed for space applications. The Guide to Plastics by the Editors of Modern Plastics Encyclopedia tabulate the resistivity of polyimides as " >1E16." W. Tillar Shugg in Handbook of Electrical and Electronic Insulating Materials, 2nd Ed. tabulates polyimide resistivity as E18.

We have performed the measurement in a simulated space environment, 1E-6 torr, 25C in our experiment lasting 24 hours. There was no clearly measured charge decay even with heating the sample to 80 degrees C. At the minimum, charge will be stored at least 19 days so that Earth and Jovian radiation will strongly charge up insulators. This result clearly demonstrates the need to take into account the proper sample conditions in a space environment.

While air molecules, humidity, and other effects help dissipate charge buildup in earth atmosphere conditions, in a space environment the charge will continue to build until harmful discharges occur. If these characteristics are overlooked and precautions not taken, using materials such as LaRC-SI and Kapton for flex circuit material or packaging in space could result in serious ESD damage to electronic circuitry.

However, it is believed that there are ways to lower the resistivity of Kapton or LaRC-SI material to dissipate charge, and yet not cause circuit cross talk. Such methods for developing dissipation will be proposed separately. The charging test methods introduced here are the proper ways to test any space material including such new or modified materials.

As a final note it might help the reader to realize that there is on-going discussion in the insulator community to explain the results of resistivity measurements using applied voltage in the classical manner. Evidence is mounting that conductive electrodes introduce charge into the sample, usually as mobile ions, which continue to move for many days and cause the applied voltage to generate a large current flow due to the mobile ions (Montanari and others, often in IEEE T. Elec. Insul). This effect is suppressed in the evacuated electron beam case where only one electrode (or perhaps none) is on the sample.